Use of Accelerated Solvent Extraction (ASE®) for Cleaning and Elution of XAD Resin

Jennifer Peterson and Sheldon Henderson, Dionex SLCTC, Salt Lake City, Utah, USA.

This article describes the procedures and the results of cleaning and eluting XAD-2 resin, using ASE. The clean resin was spiked with organochlorine pesticides (OCP) and polyaromatic hydrocarbons (PAH) standards and eluted using ASE. The results showed that ASE effectively and efficiently cleaned and dried the resin without damaging the particles, and was able to elute OCP and PAH compounds from XAD resin with good results.

Equipment is as follows: Dionex ASE 300 accelerated extractor with solvent controller, 34 mL and 100 mL stainless steel extraction cells, Dionex cellulose filters, Dionex collection bottles analytical balance (reads to nearest 0.0001 g) solvent evaporator (Turbo-Vap II®, Zymark Corporation). Materials were as follows: XAD-2 resin was Amberlite® (Supleco). Solvents were hexane and acetone (pesticide-grade or equivalent).

XAD-2 Cleaning Procedure Using ASE

Single extraction with 100% acetone followed by a single extraction with 75/25 acetone/hexane followed by two to three extractions with 50/50 acetone/hexane. ASE conditions as follows: 75 °C, one 5 min static cycle with a 150% flush and 120 s purge. Extraction cell was 100 mL and total time was 2.0–2.5 h and consumed 500 mL of solvent. This approach replaces the Soxhlet method, which requires 24 h and 1500 mL of solvents. A cellulose filter was inserted into a 100 mL cell before filling with XAD resin. XAD resin (45–50 g) was placed in the 100 mL cell and the cell's end caps were hand tightened. Three separate methods were created and saved on the ASE instrument. Method 1 was set up using 100% acetone; Method 2 using 75/25 acetone/hexane; Method 3 using 50/50 acetone/hexane. A solvent controller was used to allow the solvents to be mixed automatically for each method. All three methods use the extraction conditions listed above. Once the methods were entered and saved, a schedule was then developed. The schedule consisted of five extractions of each cell as follows: Method 1 one extraction; Method 2 one extraction; three extractions with Method 3. Because five extractions were used for the cleaning of the resin, only two 100 mL extraction cells containing approximately 100 g total of XAD resin could be cleaned per automated run.

Table 1: ASE extraction of XAD resin: recovery of OCP spike.

Compound	Recovery (% of spike)
Aldrin	87.5
α-BHC	71.8
λ-BHC	83.3
Lindane	74.7
4,4-DDD	93.0
4,4-DDE	85.4
4,4-DDT	86.7
Dieldrin	91.5
Endrin	84.1
Heptachlor	79.3
Heptachlor Epoxide	92.8

Average OCP recovery = 84.6%

XAD-2 Elution Procedure Using ASE

Single extraction with hexane/acetone (1:1) ASE conditions as follows: 75 °C, 1–5 min static cycle with a 150% flush and 120 s purge. Extraction cell was a 34 mL cell. Total extraction time was 18 min and consumed 50 mL of solvent. A cellulose filter was placed into a 34 mL cell. Approximately 16 g of precleaned XAD-2 resin was weighed into the cell. The resin was spiked with target compounds. End caps were tightened onto the cell and the cells were loaded onto the ASE 300. The samples were extracted using the elution method as described above. The extracts were then concentrated using a solvent evaporator to the desired level with a hexane solvent exchange. The extracts were analysed by GC.

Table 2: ASE extraction of XAD resin: recovery of PAH spike.

Compound	Recovery (% of spike)
Acenaphthene	86.6
Anthracene	85.8
Benzo(*a*)anthracene	99.3
Benzo(*a*)pyrene	102.9
Benzo(*a*)pyrene	100.0
Benzo(*b*)fluoranthene	102.4
Benzo(*ghi*)perylene	101.1
Benzo(*k*)fluoranthene	103.7
Chrysene	103.4
Dibenz(*a,h*)anthracene	101.4
Fluoranthene	98.9
Fluorene	92.3
Indeno(1,2,3-*cd*)pyrene	100.7
Naphthalene	81.0
Phenanthrene	99.2
Pyrene	103.4

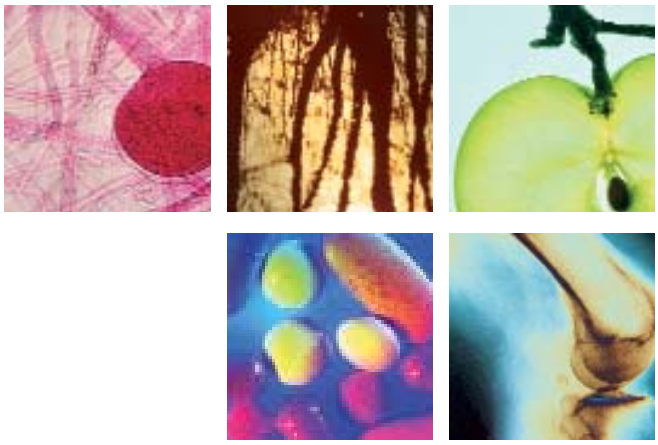
Average OCP recovery = 97.6%

Results and Conclusion

When developing a method to clean and dry the XAD-2 resin, a clean resin was defined as a flat GC–MS and GC–ECD baseline with less than 20 pg material injected onto the column. The results showed a significant decrease in resin contaminants and the resin was then considered clean. The PAH compounds were added at 100 ppm and the OCP compounds were at 10 ppb. The PAH extracts were analysed by GC–MS, while the OCP were analysed by GC–ECD. Table 1 shows the percent recoveries of the OCP standard compounds, with an average compound recovery of 84.6%. Table 2 shows the percent recoveries of the PAH standard compounds with an average compound recovery of 97.6%. ASE was able to efficiently clean and elute XAD resin, with good recoveries of PAH and OCP spiked compounds. Thanks to Edward Sverko at the National Laboratory for Environmental Testing, Burlington, Ontario, Canada for his collaboration. Thanks to American West Analytical Laboratory for environmental analysis, SLC Utah.

Reference

1. J. Yang et al., *Bull. Korean Chem. Soc.*, **20**(6), 689–695 (1999).



Cleaning and elution of XAD resin

Reader Service **272**

Available from Dionex is an application note describing the procedures and results of cleaning and eluting XAD-2 resin using ASE®. The application uses the company's Dionex ASE 300 accelerated extractor with solvent controller and other listed equipment. The results showed that ASE effectively and efficiently cleaned and dried the resin without damaging the particles, and was able to elute OCP and PAH compounds from XAD resin to good effect.

Dionex Corp., Sunnyvale, California, USA.

Human tissue proteomics

Reader Service **273**

Available from Dionex is an application note considering comprehensive 2-D nano LC–MS as an analytical tool for the separation, identification and characterization of human tissue proteomics samples. The application uses the company's UltiMate™ dual gradient nano LC system that reportedly allows for the delivery of two independent gradients down to 50 nL/min by applying a linear salt gradient in the first dimension and an acetonitrile/water/formic acid gradient in the second dimension. By using this system, the number of identified proteins almost doubled: 98 v 53 for the given human tissue sample as compared with the standard method of using salt plugs.

LC Packings — A Dionex Company, Amsterdam, The Netherlands.

DIONEX

Dionex Corporation
1228 Titan Way, PO Box 3603,
Sunnyvale, California, 94088-3603, USA.
tel. +1 408 737 0700, fax +1 408 730 9403,
website: www.dionex.com